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The magnetic and structural properties of Fe-R-B magnets were studied in the melt-spun and sintered state. The formation of $Fe_{14}R_2B$ phase including its magnetic properties (Curie temperature, magnetic moment and coercivity) and the effects of partial substitutions of iron, rare-earth and metalloid were studied in melt-spun samples. The magnetic properties of as-cast Fe-R-B powders were examined as a function of particle size. The fine powders were sintered into dense magnets and the properties of the magnets were examined over a range of temperatures. The magnetic properties of all samples (melt-spun, powders and sintered magnets) were correlated with the microstructure and magnetic domain structure in an attempt to elucidate the origin of magnetic hardening in these materials. The results of thes studies indicate that the high coercivities are due to domain wall pinning at grain boundaries in ribbons and sintered magnets and at the surface of the particles in powders.

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I. ACCOMPLISHMENTS

A. MAGNETIC PROPERTIES OF R-Fe-B-BASED MAGNETS

(i) Melt-Spun Ribbons

The magnetic properties of melt-spun Fe-R-B alloys have been examined in the whole rare-earth series. The tetragonal $Fe_{14}R_2B$ phase occurs in all alloys studied except in Eu and Yb containing alloys. The Curie temperature of this phase increases from about 170°C in $Fe_{14}Ce_2B$ to 375°C for $Fe_{14}Gd_2B$ and then decreases for the heavier rare-earths and is only 320°C in $Fe_{14}Ho_2B$. The magnetic hysteresis properties of melt-spun rare-earth boron alloys were examined and maximum coercivities (greater than 10 kOe) were obtained in Pr and Nd-containing samples. The coercivities were found to scale with the anisotropy fields which are estimated to be 40, 80 and 40 kOe for $Fe_{14}Y_2B$, $Fe_{14}Nd_2B$ and $Fe_{14}Gd_2B$, respectively.

1. Transition Metal Substitution

The effects of partial Fe substitution with Co, Mn and Ni have been examined. It is found that the Curie temperature increases steadily with Co substitution while it decreases drastically with the substitution of Mn which couples antiferromagnetically with Fe. In the case of Ni, an initial increase of T_c is observed and this is followed by a large decrease for higher Ni concentrations. Similar observations were made by Narasimhan and the results can be explained with the energy band theory. The increase in T_c for Co substituted alloys leads to smaller temperature coefficients of remanence. It is interesting to note that for higher Co substitutions the Curie temperature is increased above the crystallization temperature so that magnetic annealing is possible for these alloys. The Co, Mn, Ni substituted alloys when crystallized showed again large H_c indicating that the anisotropy of the substituted alloys is still high. In the case of Co-rich

Co-Nd-B alloys large coercive fields could also be obtained consistent with the fact that the anisotropy field of the $Co_{14}Nd_2B$ phase is comparable to that of $Fe_{14}R_2B$ phase.

2. Rare-Earth Metal Substitutions

Partial substitution of neodymium with non-magnetic (Y), isotropic (Gd) and anisotropic (Tb) rare-earths was made to understand better the effects of exchange and anisotropy on the magnetic properties of the mixed alloys. The Curie temperatures of the mixed rare-earth 14:2:1 phase are found to be an average of the Curie temperatures of the individudal rare-earth 14:2:1 phases. The increase in anisotropy by heavy rare-earth substitutions is refelected in an increase of H_c. Substitutions of Y and Gd on the other hand, lead to a reduction in coercivity.

3. Metalloid Substitutions

The $Fe_{14}R_2M$ phase is found to be formed only with boron as the metalloid with the exception of $Fe_{14}Gd_2C$ which is found to form only with Gd. The Curie temperature of this phase is found to be about 260°C with a coercivity of about 2 kOe and an anisotropy field of 14 kOe. However, the 14:2:1 phase is still formed after partial substitution of boron with other metalloids. In some cases the phase is formed even with 75% substitution of boron. This is not possible in the as-cast alloys where a mixture of 2 or 3 phases is usually observed. This again shows the high power of rapid solidification techniques in producing metastable phases that cannot be produced otherwise. The magnetic properties, T_c, M_s and H_c of the substituted alloys are found to deteriorate drastically. The larger size of other metalloids is probably sufficient to increase Fe-Fe and Fe-R inter-atomic distances leading to a decrease in J_{ex} and therefore T_c.

(ii) Powders - Sintered Magnets

The magnetic properties of powders have been examined as a function of particle size. It is found that the coercivity increases as the particle size is decreased. Optimum results were found for sizes in the range of 5-10μ. The maximum coercivity in Fe-Nd-B is found to be 4-5 kOe and it increases with Tb(Dy) substitutions.

The fine Fe-R-B powders were aligned and pressed in a magnetic field of 8 kOe. The "green bodies" were sintered at a temperature in the range of 1000-1100°C. Coercivities around 10 kOe and $(BH)_m$ around 30 MGOe have been obtained in Fe-Nd-B. The coercivity was increased with Tb(Dy) substitutions. The effects of particle size, compaction pressure, aligning field, sintering temperature and time on the final product were studied in Fe-Nd-B permanent magnets.

B. MICROSTRUCTURE STUDIES - LORENTZ MICROSCOPY

Transmission electron microscope studies on heat-treated Fe-Nd-B ribbons showed the same phases as in sintered Fe-Nd-B magnets; the tetragonal $Fe_{14}Nd_2B$ phase, a tetragonal $Fe_4Nd_{1+\varepsilon}B_4$ phase, a high Nd content phase and some a-Fe. The secondary phases are not distributed around the 14:2:1 grains but rather they are found at intersections of several $Fe_{14}R_2B$ grains. The only difference observed in the ribbons is the much finer size of the grains. The grain size in melt-spun ribbons ranges from 250-2000 Å whereas that in sintered magnets is much greater than 1μ (10-50μ). In melt-spun Fe-Tb-B alloys the $Fe_{14}Tb_2B$ grains are even finer with a size approximately 100Å. This fine microstructure is again characteristic of rapid solidification processes and leads to coercivities much higher than those observed in sintered magnets.

Lorentz microscope studies show the presence of domain walls. In the sintered magnets each grain contains several magnetic domains. In ribbons, however, the smaller grains are basically single domain while the larger ones contain several domains. The size of the domains in Fe-Nd-B is around 0.3μ and is approximately equal to that reported by Livingston for Nd-Fe-B single domain particles (SDP). In both cases domain walls end at grain boundaries and it is believed that they are pinned there. In some ribbons with very fine grain size, "multigrain" domains were also observed. These are large domains containing many grains and are explained by the interactions among the magnetic moments of neighboring grains.

C. MAGNETIC HARDENING STUDIES

In an attempt to understand the origin of the large coercive fields in these alloys, the microstructure and magnetic domain structure were correlated with the magnetic properties.

(i) Summary of results

1. The high coercivities in Fe-R-B magnets are attributed to the highly anisotropic tetragonal phase $Fe_{14}R_2B$. However, the coercivities of ribbons are much higher than those in sintered magnets.

	H_c		
Sample	Ribbon	Sintered	
$Fe_{77}Nd_{15}B_8$	>16 kOe	10-12 kOe	
$Fe_{67}Co_7Al_4Nd_{15}B_7$	>16 kOe	8-10 kOe	(Fig. 1)
$Fe_{77}Mm_{15}B_8$	8 kOe	1-2 kOe	
$Fe_{77}Tb_{15}B_8$	>145 kOe	20-30 kOe	

Co additions increase T_c but decrease H_c. Additions of Tb (or Dy) lead to an increase in H_c because of a substantial increase of the anisotropy field. The high H_c are much lower than the anisotropy field $H = 2K/M_s$ indicating a non-coherent magnetization reversal.

In ribbons the high coercivities are also obtained in samples with

composition around the stoichometric composition (presence of other phases is minimum).

2. The higher coercivities in ribbons are due to the much finer size of $Fe_{14}R_2B$ grains as compared to that of sintered magnets (a few microns, 10-50μ). In the fine microstructure samples (a few hundred Angstroms) large interaction magnetic domains are observed including many fine $Fe_{14}R_2B$ grains. In ribbons with larger grains, 2-3 magnetic domains were observed inside the grain with the domain walls ending at grain boundaries indicating domain wall pinning there. These observations roughly suggest a domain wall pinning process responsible for the magnetization reversal mechanism.

3. The initial magnetization increases slowly but gradually with magnetic field as compared to the steep increase observed in sintered magnets. This, however, is due to the random orientation of the $Fe_{14}R_2B$ crystallites (SDP) which need higher fields to align the magnetic domains. It is interesting to know that after ac or dc demagnetization the new "initial magnetization curve" lies below the one corresponding to thermally demagnetized samples. This indicates that the domain wall distribution is changed to a new state that is more difficult to magnetize showing stronger domain wall pinning.

4. The field dependence of H_c is also characteristic of domain wall pinning with a small increase at low fields, a steeper increase at medium fields and a saturation at higher fields. The smaller increase of H_c with H_a disappears in ac(dc) demagnetized samples showing again a more effective domain wall pinning in such samples.

5. The remanence curves of the ribbon samples show a similar behavior. The remanence relationship $M_D(H) = M_R(\infty) - 2M_R(H)$ is not obeyed in thermally demagnetized samples; $M_D(H)$ is the demagnetization remanence, $M_R(H)$ the initial remanence and $M_R(\infty)$ the saturation remanence.

This result indicates a non uniform domain wall pinning where the domain walls encounter different pins in the initial magnetization state than in the demagnetization state. Such behavior was expected in sintered magnets but not in ribbons with such fine microstructure. It appears, however, that after ad(dc) demagnetization the relationship is closer to being obeyed than before.

6. Based on these observation the domain wall pinning model of Gaunt was used to check the temperature dependence of H_c. According to the model for a strong domain wall pinning

$$\left(\frac{H_c}{H_o}\right)^{1/2} = 1 - \left(\frac{75kT}{4b\gamma}\right)^{2/3}$$

and for a weak domain wall pinning

$$\frac{H_c}{H_o} = 1 - \frac{25kT}{2\pi N\gamma b^2} .$$

The experimental results indicate a strong domain wall pinning.

(ii) Conclusion

All of these measurements indicate that the coercivities in Fe-R-B magnets are due to domain wall pinning at the grain boundaries. The H_c are higher in ribbons because of more effective domain wall pinning due to the many more grain boundaries of ribbons. The nature of the inhomogeneities at the grain boundaries is not yet well understood.

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IV. SCIENTIFIC PERSONNEL SUPPORTED BY THIS PROJECT AND DEGREES AWARDED

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